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**Review of Diffusion Barrier
Technology for Application
in SP-100 Fuel Pins**

M. A. Schmidt
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REVIEW OF DIFFUSION BARRIER TECHNOLOGY FOR APPLICATION IN SP-100 FUEL PINS

M. A. Schmidt and M. J. Kania

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REVIEW OF DIFFUSION BARRIER TECHNOLOGY
FOR APPLICATION IN SP-100 FUEL PINS*

M. A. Schmidt and M. J. Kania

ABSTRACT

The fuel pin design for the SP-100 reactor uses uranium nitride fuel contained in a niobium alloy (Nb-1Zr) cladding. Chemical reactions occur between the fuel and the cladding at the planned operating temperatures of 1350 to 1500 K, so a diffusion barrier is required to prevent degradation. The technology associated with diffusion barriers in similar fuel pins is reviewed to identify fabrication techniques and to summarize previous results of chemical compatibility and irradiation tests. Four fabrication techniques were identified. In previous tests, diffusion barriers have performed successfully for up to 12,000 h at cladding temperatures near 1300 K. A significant challenge remains to develop and qualify a diffusion barrier for the SP-100 mission application, which has a lifetime greater than 60,000 h.

INTRODUCTION

The SP-100 reactor is compact, lithium-cooled, and designed to operate in a space environment. The fuel element consists of sintered uranium nitride (UN) pellets, a 0.13-mm-thick (0.005-in.) diffusion barrier, and a niobium alloy (Nb-1Zr) cladding. At fuel operating temperatures of 1350 to 1500 K, chemical reactions could occur between the UN and the Nb-1Zr by the following mechanisms: (1) reduction of the UN by Nb-1Zr, (2) reaction of the Nb-1Zr with any free uranium in the fuel, and (3) reaction between Nb-1Zr and nitrogen released from the fuel.¹ A diffusion barrier compatible with both the fuel and cladding is necessary to physically separate the two and prevent the first two types of interactions.

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Previous space reactor programs developed fuel pin designs similar to that for SP-100, and the diffusion barrier technology developed in those programs can guide barrier development for the SP-100 project. Fabrication techniques, compatibility studies, and irradiation effects are reviewed to assess the performance of diffusion barriers in applications similar to SP-100.

BARRIER FABRICATION METHODS

Earlier work on lithium-cooled reactor concepts, including Pratt and Whitney's SNAP-50 program² and NASA's work on an advanced space power nuclear reactor concept,³ utilized fuel pin designs similar to those for SP-100. In the SNAP-50 program, the UN fuel pellets were contained in a tungsten-lined Nb-1Zr or PWC-11 (Nb-1Zr-0.1C) cladding and operated at a coolant outlet temperature of about 1100 K. The advanced space power nuclear reactor concept employed UN pellets in a tungsten-lined T-111 cladding (Ta-8W-2Hf) and operated at a coolant outlet temperature of about 1225 K.

Diffusion barriers used in the SP-100 program have been freestanding tubes fabricated by chemical vapor deposition (CVD).⁴ Other methods of fabricating diffusion barriers were investigated by NASA and NASA contractors. Fabrication methods investigated to produce tungsten diffusion barriers include

- (1) direct CVD of tungsten on the inner diameter of cladding,
- (2) hot isostatic pressing (HIP) of multiple wraps of wrought tungsten foil into a freestanding tube,
- (3) thermal expansion pressing of multiple wraps of wrought tungsten foil against the inner diameter of cladding,⁵ and
- (4) CVD of tungsten on the outer diameter of a mandrel followed by removal of the mandrel to form a freestanding tube.

COMPATIBILITY TESTING

Diffusion couples were investigated for the SNAP-50 program to study the UN/Nb-1Zr reaction and to identify potential diffusion barrier materials.² Couples of UN and Nb-1Zr evaluated at 1477 and 1589 K

determined the extent of the fuel-cladding reaction. After 5000 h at 1477 K, the reaction layer composed of Nb_2N and NbU alloy was 0.36 mm (0.014 in.) thick.

Tungsten, tungsten-rhenium alloy (W-26Re), tantalum, and molybdenum wrought materials were tested as potential barriers in additional diffusion couples. Tantalum reacted with UN to depths of 0.13 to 0.18 mm (0.005 to 0.007 in.) after 100 to 500 h at 1477 K. Molybdenum reacted with UN to depths of 0.003 to 0.41 mm (0.0001 to 0.016 in.) after 1000 to 6000 h at 1366 K. Tantalum and molybdenum were too reactive to be effective diffusion barriers. No reaction was observed between UN and W-26Re after 5000 h at 1477 K. Tungsten was also very stable in a diffusion couple of UN/W/Nb-1Zr with no reaction observed after 3500 h at 1477 K. With increased exposure times of 5000 and 5863 h at 1477 K, uranium penetration into the Nb-1Zr was observed to depths of 0.08 and 0.13 mm (0.003 and 0.005 in.), and a NbW phase was present in the tungsten barrier. At higher temperatures, no reaction was observed after 100 h at 1589 K, but a Nb_2N precipitate was observed in the Nb-1Zr after 100 h at 1700 K. The UN decomposed after 100 h at 1811 K.

The SNAP-50 program also tested simulated fuel pins of UN/W/Nb-1Zr in liquid lithium.² The tungsten barrier was deposited directly on the inner diameter of the cladding by the CVD process. The simulated fuel pins were tested at 1477 K for times of 1000 to 11,000 h. A diffusion layer of niobium in the tungsten barrier was observed with depths varying from 0.01 to 0.08 mm (0.0005 to 0.003 in.). A Nb_2N precipitate was dispersed throughout the Nb-1Zr cladding.

Compatibility of a UN/W/T-111 fuel-pin system at temperatures between 2075 and 2975 K was investigated using a freestanding HIPed tungsten barrier tube.⁶ Posttest evaluation revealed that for short time periods (10 min) at 2775 K and below, no evidence of fuel-barrier-cladding interaction was visible with the exception of some noticeable bonding between fuel and barrier. At temperatures above 2775 K, the tungsten diffusion barrier did not prevent reaction between fuel and cladding, and rapid decomposition of the fuel occurred. The resulting liquid uranium wet the barrier and penetrated all cracks, overlaps, and the barrier-cladding interface. It was concluded that at temperatures above 2775 K fuel decomposition occurs producing liquid uranium, which wets capsule

interior surfaces and ultimately reaches the cladding surface. The degree of barrier and cladding attack depended on the extent of fuel decomposition.

PERFORMANCE UNDER IRRADIATION

A significant quantity of data exists on diffusion barrier performance determined from postirradiation examination of in-reactor tests. Barriers produced by all of the fabrication methods listed previously (except for the differential thermal expansion method) have been evaluated after irradiation.

DIRECT CVD FABRICATION

Irradiation tests were conducted in support of the fuel-pin design utilizing CVD of tungsten directly onto the cladding. Forty-one fuel pins composed of UN/W/PWC-11 were irradiated in the Materials Test Reactor (MTR) at cladding temperatures of about 1110 to 1480 K, burnups of 0.58 to 4.58 at. % U, and times of 2635 to nearly 12,000 h (ref. 7). No defective fuel pins were detected during postirradiation examination, but examination of the 0.13-mm-thick (0.005-in.) CVD tungsten barrier revealed numerous cracks. A reaction zone between the tungsten barrier and PWC-11 cladding was noted in most fuel pins consisting of a NbW interdiffusion region and a UNb intermetallic region. This reaction zone was about 0.02 mm (0.001 in.) thick in the high-temperature (1473 K) pins and decreased in thickness with decreasing fuel-pin cladding temperature. Cracks in the barrier did not appear to have accelerated the reaction. It was postulated that uranium migrated along columnar grain boundaries of the CVD tungsten to the PWC-11 cladding.

Two fuel pins consisting of UN/W/T-111 were irradiated in the Oak Ridge Research Reactor (ORR) at cladding temperatures of about 1673 K for 5800 h to burnups of 1.75 at. % U (ref. 8). The CVD tungsten barrier for these pins was 0.08 mm (0.003 in.) thick. The performance of these fuel pins was quite different from that discussed above; one of them failed by intergranular cracking of the T-111 cladding, and the other was near failure because of cracks and cavities in the T-111. Examination revealed numerous intergranular cracks and cavities in the tungsten barrier and extrusion of the UN fuel into cracks in the barrier. In some

instances the UN had bonded to and penetrated the tungsten barrier up to about 0.015 mm (0.0006 in.). The poor performance of the T-111 cladding and the CVD tungsten barrier in this experiment was attributed to the linking together of grain boundary gas bubbles under tensile stress.^{9,10}

FREESTANDING HOT ISOSTATIC PRESSING FABRICATION

Six fuel pins utilizing a UN/W/T-111 fuel-barrier-cladding design were irradiated in the ORR at about 1265 K cladding temperature for approximately 10,000 h to a burnup of about 3 at. % U (ref. 11). The barrier material in this pin design was a 0.08-mm-thick (0.003-in.) freestanding wrought tube fabricated by hot isostatic pressing. The postirradiation examination revealed major cladding cracks in two fuel pins resulting from hydrogen embrittlement of the T-111. All the barriers had some cracks, but no fuel-barrier or barrier-cladding reactions were evident. It was concluded that even though cracks would occur in the liner during irradiation, they should not cause compatibility problems provided UN did not contact T-111 cladding.

FREESTANDING CVD FABRICATION

The first series of SP-100 irradiation experiments (SP-1, SP-2, and the planned SP-3) have as part of their test matrix a UN/W/Nb-1Zr or UN/W/PWC-11 fuel-pin design in which the barriers are 0.13-mm-thick (0.005-in.) freestanding tungsten tubes fabricated by the CVD process. The SP-1 and SP-2 irradiation experiments are being conducted in the EBR-II reactor at cladding temperatures of 1300 and 1500 K to fuel burnups of 0.8 to 3.8 at. % U.

Postirradiation examinations performed on the first phase of the SP-1 test indicated that no fuel-pin failures occurred; however, interactions between the UN fuel and the tungsten barrier and between the barrier and the Nb-1Zr cladding did occur.^{4,12} The barriers showed evidence of intergranular cracking of the CVD tungsten similar to that reported in ref. 8. A NbW solid solution was identified by microprobe analysis at the barrier-cladding interface. Microprobe analysis also indicated the presence of free uranium at the fuel-barrier interface, probably due to

the high operating temperature (1960 K surface) of the hypostoichiometric UN fuel. Molybdenum may have accelerated the UN dissociation. It was present as an impurity both in the tungsten barrier and on the surface of the UN fuel pellet. Ruthenium, a metallic fission product, was also found deposited between the barrier and the cladding material. No interactions were observed between the UN fuel and the Nb-1Zr cladding; the tungsten tube functioned as a suitable diffusion barrier under the exposure conditions.

SUMMARY

Compatibility tests evaluated Ta, Mb, W, and WRe alloy (W-26Re) as potential diffusion barriers for UN and Nb-1Zr. Tantalum and molybdenum were incompatible with UN at temperatures of 1477 and 1366 K, respectively. The WRe alloy did not react with UN after 5000 h at 1477 K. Tungsten was quite stable, but formed a 0.13-mm (0.005-in.) reaction layer with Nb-1Zr after 5863 h at 1477 K in a diffusion couple. This reaction layer was only 0.08 mm (0.003 in.) thick in a simulated fuel pin tested in liquid lithium after 11,000 h at 1477 K.

Irradiation experience with diffusion barriers is summarized in Table 1. Fuel pins have been tested with tungsten as a diffusion barrier between UN fuel and three different cladding materials: Nb-1Zr, PWC-11, and T-111. Tungsten barriers have consistently cracked during irradiation, regardless of the fabrication method or the cladding type. Barrier-cladding interactions were observed for the niobium alloy claddings, Nb-1Zr and PWC-11, but were not present with the T-111 cladding.

Table 1. Summary of irradiation experience with tungsten barriers in UN fuel pins

Clad	Barrier type	Cladding temperature (K)	Time (h)	Burnup (at. % U)	Cladding failure	Observed interactions ^a			Reference
						Barrier-cladding	Barrier-fuel	Fuel-cladding	
PWC-11	Direct CVD W	1,100-1,480	2,635-12,000	0.58-4.58	0 of 41	yes	---	yes	7
T-111	Direct CVD W	1,673	5,800	1.75	1 of 2	no	yes	no	8
T-111	HIP W tube	1,265	10,000	2.8	2 of 6	no	no	no	11
Nb-1Zr	CVD W tube	1,500-1,600	2,400	0.8	0 of 2	yes	yes	no	4

^aCracked barriers were found in all experiments.

Interactions between the tungsten barrier and the UN fuel were observed at cladding temperatures above 1500 K but not at 1265 K.

Despite the postirradiation condition of the barriers, the performance of the fuel pins clad with niobium alloys was good. No failures of either Nb-1Zr or PWC-11 cladding were observed, although some fuel-cladding interactions were identified in ref. 7. The performance of the diffusion barriers was therefore adequate for the conditions. Failure of the T-111 cladding was observed in refs. 8 and 11, but no fuel-cladding interactions were present. The diffusion barrier served its purpose - to prevent direct chemical interaction between UN and T-111 - but did not prevent failure of the T-111 cladding.

The performance data available on tungsten diffusion barriers fabricated by several methods can be used to estimate their value for current SP-100 reactor operating conditions. The data clearly demonstrate an operating lifetime of 10,000 to 12,000 h for a fuel-barrier-cladding design of UN/W/PWC-11, fuel operating temperature of 1400 to 1500 K, cladding temperature of about 1300 K, and peak burnup in excess of 4 at. % U. Longer lifetimes may be likely, but the experimental data base only extends to 12,000 h. The UN fuel was fabricated by a procedure different from the present SP-100 reference procedure, so revalidation of the fuel system performance is required.

The barrier fabrication methods discussed in this assessment were direct CVD of tungsten on the inner surface of cladding and HIPing of thin tungsten foils into freestanding tubes. For the HIPing method, a UN/W/T-111 fuel-pin design was used. In both methods, the barrier material was 0.13-mm-thick (0.005-in.) unalloyed tungsten. It should be noted that only small-scale pins 150 to 300 mm long (6 to 12 in.) were tested.

RECOMMENDATIONS

A significant challenge remains to develop and qualify a barrier material and select a fabrication method adequate for the SP-100 mission application. The barriers employed in past programs were designed for

systems operating at lower fuel and cladding temperatures and for reduced system lifetimes. Further, the UN fuel was fabricated by a method different from that used for SP-100.

Developing an effective diffusion barrier technology will require characterizing a number of candidate fabrication methods through activities closely integrated with the fuel development efforts and the fuel system irradiation experiments. Currently, only freestanding CVD tubes have been evaluated. Barriers fabricated by direct CVD inside the cladding or by HIPing of wrought foils are also of interest. Materials besides tungsten, such as Re or WRe alloys, should also be evaluated. Fuel-pin performance could be improved by removing the barrier-cladding gap during fabrication, either by direct CVD onto the cladding, HIPing of the cladding onto the barrier, drawing the cladding onto the barrier, or by some other method producing a bonded (duplex) barrier-cladding tube. The barrier fabrication method selected will require scale-up to produce full-size fuel pins.

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